



## Criminalistics Section - 2016

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### **B48 Identification and Separation of Nitrate Esters Using Both Liquid Injection Gas Chromatography/Mass Spectrometry (GC/MS) and Total Vaporization Solid Phase Microextraction (TV-SPME) GC/MS**

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After attending this presentation, attendees will understand the value and need for a singular method that allows for the identification and separation of various nitrate esters found on exploded devices. TV-SPME/GC/MS is a new technique that has several benefits over traditional liquid injection.

This presentation will impact the forensic science community by providing a singular method of analysis for nitrate esters, saving both time and money as well as increasing sensitivity.

Given that most laboratories utilize a liquid injection procedure, a method has been developed using this technique; however, a novel technique based upon TV-SPME/GC/MS has also been used with great success in identifying compounds of interest. This novel technique has the inherent benefit of having a much lower limit of detection and can therefore allow for identification with less sample preparation and small sample sizes. This is of great interest as the explosive used in a device can be determined from smaller samples recovered from a post-blast site.

The detection and identification of post-blast residues is an important part of an explosives investigation; however, various methods must be used in determining the type of explosive that was used in the device. This study sought to determine if a single method could be found to separate and identify the various nitrate ester explosives as well as their degradation products. It was found that GC/MS could be used to differentiate a number nitrate esters such as Nitroglycerin (NG), Ethylene Glycol Dinitrate (EGDN), Pentaerythritol Tetranitrate (PETN), Erythritol Tetranitrate (ETN), Pentaerythritol Trinitrate (PETriN), 1-nitroglycerin, 2-nitroglycerin, 1,2-dinitroglycerin, and 1,3- dinitroglycerin.

Nitrate esters fragment identically within a mass spectrometer; therefore, having a true separation using a GC program prior to entering the MS is vital. Several variables were investigated using liquid injection, such as inlet temperature, flow rate, and inlet temperature ramp speed. In addition to liquid injection, a novel method was developed using TV-SPME/GC/MS. TV-SPME is a process that is most akin to immersion SPME. In immersion SPME, a two-phased system is present between the liquid containing the analyte and the SPME fiber. In TV-SPME, heat is used to drive all of the analyte into the gaseous phase. By taking a small amount of liquid that can be totally vaporized into the gaseous phase, one is able to create a two-phase environment between the analyte in the gaseous phase and the SPME fiber. This has the benefit of increasing sensitivity and decreasing sample preparation. Throughout this study, the following parameters were optimized: fiber type, incubation temperature, and multi-stage inlet temperature ramp. This method was able to separate and identify PETN, EGDN, NG, and ETN and the detection limits for these compounds was as low as 50 parts-per-trillion. Post-blast debris from the initiation of PETN-based plastic explosives was also analyzed.

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#### **Nitrate Esters, Explosives, SPME**